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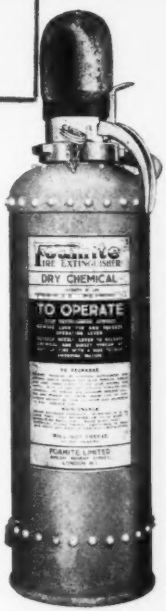

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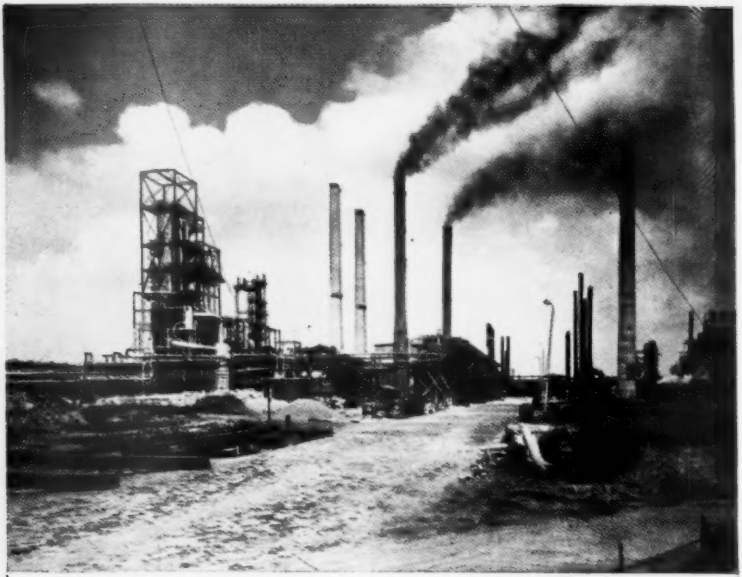

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PETROLEUM IN THE PAINT, PLASTICS AND RUBBER INDUSTRIES*

By A. J. GOODFELLOW (Associate Fellow) †

Until about 30 years ago petroleum was refined by conventional methods to yield, as distillation products, motor spirit, kerosine, lubricating oils, fuel oils, and bitumens. At that time the materials within the scope of this paper would have been limited to such distillation products as straight-run gasoline fractions, bitumen, and carbon black.

The development of controlled cracking and reforming has provided chemical bricks in the form of the simple gases methane, propane, butane, ethylene, propylene, butylene, and butadiene from which a vast array of synthetic products may be derived.

* Summary of a paper read to the London Branch, Institute of Petroleum, November 20, 1950.

† Carless, Capel and Leonard Ltd.

THE PAINT INDUSTRY

It has been said that one of the blessings of civilization is paint. Man has used paint in one form or other for decoration and protection ever since he lived in a community and paint making must rank as one of the earliest arts. A modern decorative oil paint consists of a combination of a solid constituent, the purpose of which is to obscure the surface and provide colour and decorative effect, with a liquid constituent the function of which is to bind the solid permanently to the surface and protect it from decay.

The solid constituent amounts to between 60 and 80 per cent of the

finished paint and usually consists of a white and coloured pigment together, perhaps, with a small percentage of inert extender. The only pigments provided by the petroleum industry are the blacks, e.g., carbon black used to the extent of about 2,000 tons per year by the paint industry[1] and bitumens used in making black varnishes. The latter are valued for their resistance to water, but a defect is that they cannot be over-painted, since the bitumen bleeds into the next coat applied.

The liquid medium in its simplest form consists of a natural drying oil, the commonest being linseed oil, with added driers to regulate the drying of the oil and a volatile solvent to adjust the consistency for application. In more complicated paints the natural drying oil is replaced by heat-treated oils and varnishes.

In paints containing drying oils the essential drying after application to form a hard protective coating is mainly due to the chemical changes which take place in the oil on exposure to air. The oxidation is catalysed by the incorporation of dryers, one of the most important and effective types being the metallic naphthenates, for example, cobalt naphthenate. Naphthenic acids occur in the gas oil and kerosine fractions of certain petroleums and prior to 1930 were regarded as waste products.

Although the petroleum industry is not yet a source of drying oils, it does provide an extender and substitute for the natural product. During the war time shortage, when careful investigation of substitute possibilities was necessary, it was found that the aromatic petroleum extracts, obtained as a by-product of oil refining, could be used in all types of paints. Although not truly drying oils they were found to be compatible with natural drying oils and paint thinners, and could be used in concentrations of up to 20 per cent with linseed oil without impairing the performance of the paint.

There are patents[2] for the preparation of unsaturated compounds of high molecular weight, and this field offers promise for future development of synthetic drying oils.

Volatile solvents or thinners are used to lower the viscosity of a paint and to facilitate its application. After application the thinner is no longer required and it is essential that it should be completely volatile. Prior to 1885 turpentine was the only volatile solvent used in the paint industry, but in that year Samuel Banner introduced his patent turpentine, a petroleum distillate. For many years it was regarded as a cheap adulterant for turpentine, and as a "turpentine substitute." Its merits as a paint thinner gradually were recognized, and it is now universally known as white spirit.

The present consumption of white spirit in the paint industry is about 30 million gallons a year, compared with 1.5 million gallons of turpentine, a definite reversal of the original order of importance.

The gloss obtainable with oil paint is limited by the paint not flowing over the surface sufficiently to eliminate brush marks. To obtain a smooth finish it is necessary to increase the flowing properties of the medium by the use of heat treated oils, whilst the brilliance and hardness of the surface is materially increased by the addition of varnishes.

The making of durable oil varnishes was originally based on combining the hard natural fossil resins with linseed oil, the resins being modified by heat treatment or esterification to render them compatible with the drying oils and solvents. The introduction of synthetic resins has simplified the manufacture of oil varnishes and increased their range of usefulness. Petroleum is now a basic raw material for the resin industry.

The thinners used in the synthetic varnish industry must be good solvents for the resin in use and in general

hydrocarbons with a high aromatic content are required. The aromatic content of modern white spirit is only about 15 to 20 per cent and this percentage is increased by admixture with the aromatic hydrocarbon fractions obtained from the coal tar industry. The fractions so used are xylole and medium and heavy naphthas. Petroleum aromatic fractions may be used similarly.

Hewitt[3] summarized the essential features of an ideal film forming material as:—

(1) Ease of application out of solutions in cheap non-objectionable solvents;
(2) Greatest possible speed of drying compatible with good film forming properties;
(3) Greatest possible resistance to physical and chemical agents;
(4) Good colour and colour retention;
(5) Good glossing properties.

The ideal surface coating has not yet been developed but a remarkable progress has been made during the last twenty years in the development of synthetic resins, and we are now on the threshold of a further period of development through products originating from petroleum.

The synthetic coatings may be conveniently classified in two groups.

Group 1—highly polymerized resins applied to a surface through the medium of a solvent and deposited by evaporation. No essential change takes place in the structure of the polymer resins and the film retains its solubility in the original solvent. This group includes cellulose lacquers, vinyl resins, polystyrene resins, etc., and these surface coatings are usually applied by spraying.

Group 2—the high molecular structure in embryonic form, which, after application, polymerizes and is converted to an insoluble film. This group includes the phenolic resins and the alkyds.

The alkyds which have been developed

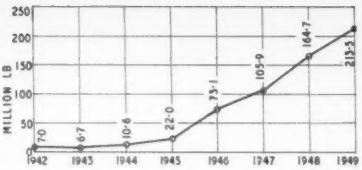

Fig. 1. Polystyrene production (U.S.) population.

rapidly since 1930 are obtained by the esterification of polyhydric alcohols, e.g., glycerine, with polybasic acids such as phthalic acid.

It has been shown how the aliphatic white spirit and aromatic naphthas are essential to the paint industry. So also are newer solvents now being marketed —the chemical products from petroleum.

The possibility of using cellulose derivatives in surface coatings was first recognized by Alexander Parkes, who suggested the use of nitro cellulose and the addition of resins. The discovery of amyl acetate in 1882 provided a solvent of the right evaporation characteristics and, with camphor as a plasticizer, the cellulose lacquer industry became a probability. However, it was not until other solvents such as butyl acetate became freely available that large scale production became practicable. A cellulose lacquer consists usually of:—

(1) A cellulose ester; (2) a resin; (3) a high boiling solvent; (4) a medium boiling solvent; (5) a diluent; (6) a plasticizer; and (7) a pigment.

The desired properties are obtained by varying the constituents to give the optimum result.[4,5]

The petroleum industry is now making a major contribution to the cellulose paint industry, since not only does it provide a pigment (carbon black) and the diluents (special boiling point fractions, and aromatic fractions) but also the requisite true solvents and some plasticizers. The petroleum chemical industry manufactures ethyl and *iso*-propyl alcohols, ketones and esters,

carbitol, cellosolve, etc., as solvents, and such compounds as glycol phthalate, tri-ethylene glycol, triacetin, etc., as plasticizers.

These are but a few of the many solvents available for cellulose and synthetic lacquers, and which are produced to a very high degree of purity.

As the combined percentage of solvent, diluent, and plasticizers for most spray lacquers amounts to about 80 per cent of the total, it is obvious that the paint industry will be a larger consumer of the newer products from petroleum.

The Plastics Industry

Delafield, chairman of the British Plastics Federation, recently described the plastics industry as "a world in itself, young and still not fully explored and yet part of a greater chemical universe." If the discovery of celluloid, by Alexander Parkes, in 1865, is taken as the birth of the industry it may not seem quite so young, but in the early days plastic products were sold and accepted by the public as "artificial substitutes," e.g., tortoise-shell, ivory, etc. As an industry proper it dates from about 1928. At the end of the first world war, Britain's production of plastics was not more than a few hundred tons, such products as synthetic resins being used mainly in the paint and lacquer industries. Growth of the gramophone and radio industries led to the wide use of cheap, easily moulded, and highly insulating base plate materials and to increased production of moulding powders of the phenol type.

The incentive to develop synthetics of the rubber type was not great in the U.K. since Britain controlled the greatest supplies of natural rubber and the lack of cheap electric power mitigated against development of syntheses based on acetylene. On the other hand, British research was largely responsible for acrylate thermoplastics, i.e., Perspex, for polyvinyl-chloride polymers, for terylene, and for polythene.

In 1945 journalists were writing of a coming plastics age, although one expert stated that to replace 5 per cent steel and 10 per cent timber with plastics based on coal, would necessitate the annual carbonization in Britain of four times the world output of coal.

To-day the plastics industry covers both the producers of synthetic resins and moulding compounds, and also those who mould and fabricate many articles in everyday use. Both groups are large consumers of petroleum fuels and lubricants.

The war-time and post-war problems of the U.K. plastics industry were the provision of adequate productive capacity, and the procurement of adequate raw material supplies[6] at prices competitive with those of other countries. The main weight of research effort in Britain is directed to solving the latter problem.

At present the spotlight is on oil as a store-house of an almost unlimited variety of chemicals. Experienced oil refining groups have combined with chemical and plastics manufacturers in erecting oil cracking plants in which synthetic chemicals are the ultimate goal. The major effort is directed according to the general trading bias of the companies concerned, the final products being organic solvents, thermosetting or thermoplastic materials or fibres.

The reason and need for the petroleum industry entering the plastics field is that, apart from the questions of economics and technical simplicity, the rapid growth in every field has been such that it may well have outstripped the available supply of raw materials.

Until recently, benzene, which is of vital importance to the plastics industry, was derived almost entirely from coal. It is the raw material for the production of synthetic phenol required for the phenol formaldehyde type of plastics and, reacted with ethylene, it produces monostyrene after dehydrogenation. The growth of the use of polystyrene in

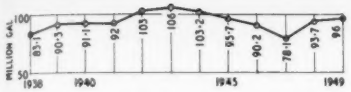

Fig. 2. U.K. production of crude benzole.

the United States is shown in Fig. 1 and there are considerable potentialities in Britain. Benzene is also the basic material in the production of adipic acid and hexamethylene diamine, which are condensed together to give nylon.

Crude benzole production in Britain (Fig. 2) was 96¼ million gallons in 1949 of which motor benzole took 58¾, 90's benzole 4½, and pure benzene 3½ million gallons. During 1949 the plastics industry used 1¼ million gallons for the ultimate production of phenolic plastics and in two years time about 3¾ million gallons or about 4 per cent of the entire production will be used, i.e. 7–8 per cent of the actual benzene present. As it takes about 350 gallons of benzene to make a ton of styrene the demand for benzene will increase.[7] No dramatic increase in benzole manufacture can be expected from coal carbonization, but extra supplies will be coming forward from cracking of petroleum. The expansion of the petroleum cracking industry will make available many raw materials.

Ethylene—Ethylene is a basic material of increasing value to the plastic industry. In 1933, during unsuccessful attempts in the I.C.I. laboratories, to combine ethylene with benzaldehyde under a pressure of 30,000 p.s.i. at 170° C, a small quantity of white waxy solid was produced. This proved to be a polymer of ethylene with valuable electrical properties, light in weight, and almost inert to chemical attack. It now finds use in submarine cables, in chemical and table ware, and in crepe sheet, and Fig. 3 illustrates the rapid increase in production. Polyethylene is manufactured under British-owned patents and the I.C.I. Wilton plant is expected to produce a polymer cheaper and competitive with any polythene produced in the world.

By progressively replacing the hydrogen in ethylene with chlorine or fluorine, a further series of polymers of remarkable chemical inertness has been produced. Thus, poly-tetra-fluorethylene which, although difficult to manipulate, gives such a remarkable industrial service that a raw material price of £5 per pound is justified.

In America the largest consumption of ethylene is for the manufacture of ethyl alcohol, the growth being from 9 million gallons in 1935 to 100 million in 1948, when 66.7 per cent was synthetic.[8] Ethyl alcohol is not only an important solvent but also the starting off point for many chemicals, such as acetaldehyde, acetic acid, ethyl acetate, etc., which find secondary uses in the plastics industry.

From propylene and butylene are obtained the corresponding alcohols and derivatives, solvents which are so useful to the paint industry. Acetone is synthesized by the catalytic dehydrogenation of *iso*propyl alcohol and, apart from its usefulness as a solvent, is the starting point for the manufacture of "Perspex." The acetone is first converted into acetone cyanohydrin by treatment with hydrogen cyanide. The cyanohydrin is then treated with sulphuric acid and methyl alcohol and on subsequent dehydration gives monomeric methyl methacrylate. This liquid after purification is changed into the well known plastic by polymerization, the method depending on the type of polymer required. In addition to its transparent form, Perspex is also

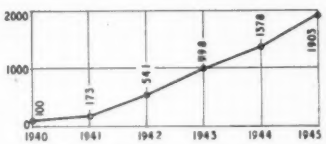

Fig. 3. Polyethylene production index (U.K.)

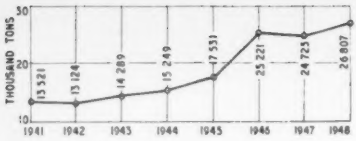

Fig. 4. U.K. production of phenolic and cresylic moulding powders.

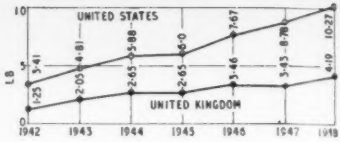

Fig. 5. Plastics production per head of population.

available in colours. Polymethyl methacrylate, like polythene, was not made in large quantities until the outbreak of war. From 1941 to 1944, the production trebled and in 1948 production in the U.K. was 4000 tons, almost equal to the American output.[9] It is estimated that 2800 tons of acetone are needed to produce this amount of the polymer.

The paint industry is a large consumer of synthetic resins, the U.K. production of which rose from 16,000 tons in 1941 to 45,000 tons in 1948.[10]

Phenolic and cresylic moulding powder production rose from 13,500 tons to 26,800 tons over the same period,[9] (Fig. 4), while polyvinyl plastics rose from 745 tons in 1943 to 17,000 tons in 1948.

Petroleum is not used in the U.K. for the production of the polyvinyls, which are based on acetylene, or of the synthetic resins based on glycerol, phenol, cresylic acid, etc. In the U.S.A. manufacturing processes operate for the production from petroleum of ethylene glycol and maleic anhydride for the preparation of alkyd resins, phenol for moulding compositions, and vinylidene chloride monomer for polymerization to a useful plastic, whilst the commercial production of allyl chloride and allyl alcohol from petroleum has not only resulted in the manufacture of glycerol, but has opened up a vast new field of solvents and chemicals.[8]

Comparing the relative plastics production per head of population (Fig. 5) it is seen that although the American industry has expanded at about the same rate as the British and is seven times as large, the production per head of population is only 2½ times the British figure.[9,11]

The Rubber Industry

Ever since Mackintosh discovered the solvent properties of petroleum for rubber, the rubber industry has been one of the largest consumers of petroleum solvents. In proofing, the rubber is mixed into a dough with suitable pigments, fillers, and accelerators and then spread on to the fabric. The solvent is evaporated and the rubber vulcanized by treatment with sulphur compounds.

Rubber tyres are built up from successive layers of fabric impregnated with rubber, the tread is then fitted and the whole tyre consolidated by vulcanization in a mould. Certain articles are manufactured by dipping a former into a rubber solution. The solvent evaporates and a layer of rubber of the desired thickness is gradually built up. Vulcanization completes the process.[12]

For the purposes described one or other of the special boiling point fractions or petroleum rubber solvent 100–160 is usually employed. These fractions are also used in the preparation of rubber adhesives which have the property of uniting at ordinary temperatures to form a bond of great strength.[13] The reason is that the surfaces exhibit the property of dry tack.

The formulation of a rubber solution is varied to suit the job, and the viscosity may be controlled in relation to the solid content by the degree of mastication of the rubber. The drying time

depends on the choice of the petroleum solvent.

Examples[14] of the use of these solutions are in the motor car industry, where nearly 4 gallons of solution are used for the upholstery, etc., of each car, the boot and shoe manufacturers and repairers, and in more recent years in the manufacture of self-sealing fuel tanks and tank water-proofing. During the last war every vehicle marshalled to take part in the military landings carried a kit containing patches and tubes of rubber solution for waterproofing the vehicles and enabling them to land through water. Nearly ten million gallons of special boiling point spirits are consumed annually mainly by the rubber and paint industry.

Practically the whole of the world's carbon black production is from natural gas and petroleum and until recently Britain was entirely dependent upon the United States for its supplies. The shortage of this product during 1947 showed how indispensable carbon black was, particularly to the rubber industry. Tyres made from natural rubber are useless, they become brittle and wear out quickly. Carbon black is an ideal compounding ingredient for rubber, its reinforcing qualities being roughly proportional to the amount incorporated up to about 40 per cent by weight. For special purposes loadings of up to 65 per cent are used. Cadman[15] records that the value of carbon black was fully realized by the tyre industry in 1920. Tyre treads usually contain 45 parts of furnace or channel black per 100 parts of rubber, inner tubes about 35 parts of furnace black, and carcases about 25 parts furnace black per 100 parts of rubber. Tyre life has increased from 2000 miles in the very early days to about 5000 miles with other reinforcing pigments and to about 30,000 miles with carbon black. Synthetic rubbers need a higher percentage of carbon black than the natural rubber and compound better with the furnace type of black. This has meant first that the tonnage of synthetic rubber was expanded during the difficult war years and, secondly, that much work on the manufacture of furnace blacks from a variety of fuels has led to the erection of producing plants in Britain.

The importance of the petroleum industry to industries consuming carbon black can be measured by the growth in consumption. The total quantity of carbon black exported from the U.S.A. to the U.K. was about 44 million lb in 1938, 62 million lb in 1939, and 77 million lb in 1940. About 80 per cent of this went to the rubber industry. During the latter part of 1948 several organizations announced their plans for making carbon black of the reinforcing type in Great Britain.[16]

The rubber industry uses a large number of organic compounds, some of which are based on petroleum. For example, many plasticizers are based on petroleum asphalt whilst aromatic extracts, paraffin wax and naphthenic acids find specialized uses.

The information regarding the synthetic rubber industry is derived mainly from *The Times Review of Industry* (June, 1950) and refers to United States production. The U.S. produced 215,000 tons in 1943, more than three times as much in 1944, and in 1946 the output of 1,176,000 tons exceeded the quantity of natural rubber consumed by the whole world in 1939. Four types of material were made :—

1. Buna-S which consists of about 75 per cent butadiene and 25 per cent styrene;
2. Buna-N consisting of 75 per cent butadiene and 25 per cent acrylonitrile. Butadiene may be made from petroleum, natural gas, or grain alcohol, styrene from petroleum or coal tar, and acrylonitrile from petroleum and nitrogen;
3. Butyl, made wholly from the petroleum derivatives *iso*butylene and isoprene;

4. Neoprene made chiefly from acetylene and chlorine.

During 1950 a new synthetic rubber called poly-butadiene or P.B. rubber has been developed. Limited road tests indicate that it is superior to GR-S synthetic. The raw material is butadiene and substitution of this rubber for GR-S will relieve, to some extent, the requirements of styrene and hence benzene.

Nearly 85 per cent of the war-time output was of the Buna-S type and two thirds of the 700,000 tons of butadiene needed annually were obtained from petroleum.

It was not possible to find from any source other than petroleum the enormous quantities of hydrocarbons required for this purpose. The war precipitated the use of hydrocarbons to create an organic chemical industry which, in its first few years of existence, left the "Classical" chemical industry far behind.[17]

Conclusion

The impact of the petroleum-chemical industry has been felt more by the three industries discussed than by any others. A close study of their requirements has been directly responsible for the development of the petroleum chemical projects in Britain, whilst they are also large consumers of traditional petroleum products.

In a lecture on "Petroleum Chemicals, 1950 and 2000 AD", Dr G. Egloff predicts production growth in the U.S.A. as follows:—

	1950	2000
Petroleum Chemicals	*Thousand tons*	
Raw materials from petroleum and natural gas ...	2000	7000
Finished products ...	5000	21000
Synthetic ethyl alcohol ...	250	550
Synthetic glycerol ...	15	44
Aromatic hydrocarbons from petroleum ...	330	3000
Chemical Products partly from Petroleum		
Synthetic rubber (50% petroleum base)	400	1800
Plastics 60% petroleum base	800	3300

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★ ★ ★

Lubrication of Steam Turbines.—Latest booklet issued by Shell Petroleum Co. Ltd, deals with the lubrication of steam turbines. It contains over 100 pages and in 14 chapters gives much information on the subject. Copies are obtainable from Shell-Mex and B.P. Ltd, Shell-Mex House, Strand, London, W.C.2.

Air Separation Plants is the title of an illustrated descriptive booklet recently issued by Petrocarbon Ltd. Four types of plant are described: a small plant for liquid nitrogen, a small plant for gaseous oxygen, a medium plant and a larger plant for oxygen or nitrogen.

CORRESPONDENCE

A Hidden Hazard

To the Editor, *I.P.Review*,

Sir: The following occurrence may be of interest to readers of the "I.P. Review."

The writer had occasion recently to purchase some rags, for use on car maintenance and similar domestic tasks.

Whilst wiping down some spilt petrol with one of these rags (a portion of an army denim suit) it was found that, tucked away in the remains of what had been a pocket, were a couple of unused non-safety matches. Despite the friction to which the rag had been subjected the matches did not happen to light (although they were afterwards found to be ignitable in the usual way) and nothing came of the incident.

As, however, bulk purchase of rags from commercial sources is a common procedure in refineries the existence of such a risk, which is remote and might well be overlooked, is perhaps worth recording.

V. Biske.

5 Kingsmead, Upton Lane, Chester.
September 1, 1951.

PERSONAL NOTES

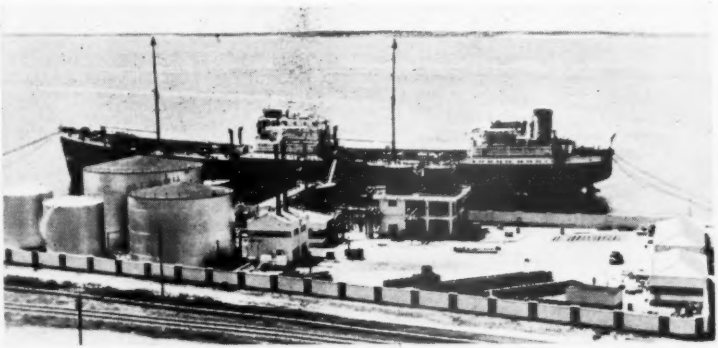
Leonard Sinclair, M.Inst.Pet., has been elected to the board of directors of Esso Transportation Co Ltd. Mr Sinclair became chairman and managing director of Esso Petroleum Co last June.

Philip A. Singleton has been appointed a director of Monsanto Chemicals Ltd. His first appointment with the organization was in 1940, when he joined the Merrimac Division of Monsanto Chemical Co., St. Louis, U.S.A.

Vernon Harbord, F.R.I.C., F.I.M., has been elected President of the Institution of Mining and Metallurgy for the 1952-3 session.

Sir Felix Pole has become chairman of the board of the Pulsometer Engineering Co in place of Mr Eliot Hodgkin, who has resigned.

The Minister of Fuel and Power has appointed **W. G. C. Reed** to be a member of the Oil Consumer's Council. A member of the Board of Management, National Chamber of Trade, he replaces **A. F. Hopkinson,** who has taken up residence abroad.



The tanker Labiosa *is seen here discharging bulk bitumen at Setubal, Portugal, where an installation of 6000 tons capacity was recently opened. This is the second depot of its kind to be completed in Europe for Shell and associated companies in a programme whereby bitumen is supplied in bulk direct from Caribbean refineries. Three specially converted 9000-ton tankers are being used for this operation.*

REPORT OF WEIGHTS AND MEASURES COMMITTEE

In 1948 the Board of Trade set up a committee "to review the existing weights and measures legislation and other legislation containing provisions affecting weights and measures and the administration thereof, and to make recommendations for bringing these into line with present day requirements."

The Committee has recently published its report in a comprehensive document of 147 pages (Cmd. 8219, HMSO, London, 3s. 6d. net). The portions which are of most interest to the petroleum industry are paras 164–169 in Chapter 8 on Equipment to be Brought under Control in Part II : Weighing and Measuring Equipment, and paras 370–1 in Chapter 16—Commodities other than Food requiring Special Treatment—in Part III : Short Weight and Measure.

These paragraphs are reproduced below :—

Bulk Liquid Measuring Instruments

164. The Board of Trade is empowered to make regulations covering the verification and inspection of liquid measuring instruments under the 1904 Act ; but no regulations have been promulgated to cover instruments used for the measurement of individual quantities in excess of 20 gallons. There is thus no official supervision of the initial accuracy or of the satisfactory maintenance of instruments used for delivering quantities above this figure; and the use or possession for use of a false or unjust instrument of this type is not an offence under the Weights and Measures Acts, nor can fraud in its use be punished under them.

165. Our attention has been drawn to the fact that the tank wagons which are normally used for delivering fuel oils to garages, etc., constitute a measuring instrument in that sales are based on a measurement, by means of a dipstick, of the amount contained in or discharged from the tank wagon. Most tank wagons are divided into a number of compartments; and each compartment, together with the dipstick which has been calibrated to give readings of the quantity in it, constitutes a measuring instrument which is unquestionably in use for trade in these cases. There are in existence about 4,700 tank wagons delivering petrol in this way, comprising some 16,000 compartments which may contain from 200 to 500 gallons each; and the normal practice of the petroleum companies is to deliver at least one complete compartment to each retailer to avoid the difficulties inherent in measuring split loads.

166. It has been represented to us that the dipstick method of measurement is unsatisfactory in that accuracy can be prejudiced by the existence of swirl in the liquid caused by the movement of the wagon prior to discharge or by the stationing of the wagon during discharge, on a slope, and also that it lends itself to fraud in that the reading can be appreciably affected by handling the dipstick in other than in the correct manner. It has in fact been suggested to us that some form of bulk flowmeter affords the only satisfactory method for this type of transaction. On the other hand, the association representing the petroleum companies has claimed that the dipstick method can be accurate to within something like 0·16 per cent; that, in accordance with Home Office Regulations, each compartment has a dip-tube which serves to minimize the effect of swirl; and that the possibility of manipulation is diminished by the use of a T-shaped dipstick which rests on a cross-bar when the dip is being made and by the encouragement which the companies give to the retailers to supervise the process of measurement. It is further claimed that flowmeters

which are operated by gravity give far less accurate readings than dipsticks in the hands of trained operators and that no flowmeter has been marketed which would stand up to the strain of the road travel involved in deliveries. On these grounds it is held that the present method is as satisfactory as is possible under the circumstances.

167. We accept this view. The present method of measurement is by no means ideal and only supervision by the receiver can eliminate all possibility of careless or deceitful practice; but we do not see that any other could be compulsorily introduced in the near future without considerable dislocation to the trade. We are nevertheless in sympathy with the strong representations that have been made to us by the retailers of fuel oils that, if this method is to be continued, the purchaser should have some guarantee that the equipment has been subject to official verification; and the petroleum companies have indicated that they would have no objection to their tank wagons and dipsticks being brought under full Weights and Measures control.

168. We therefore recommend that the patterns of existing and future types of wagons and dipsticks should require to be approved by the Board of Trade in the manner outlined in the preceding Chapter; that all wagons and dipsticks at present in service for bulk sales of fuel oils and all subsequent replacements or additions should be required to be verified and stamped by an inspector; and that, after a suitable interval, the use of an unstamped measuring instrument of this sort or the use of a false or unjust instrument or fraud in its use should be an offence. We have received information from the Standards Department of the Board of Trade indicating that verification of the tank wagons and dipsticks now in existence would take about one year to complete and that, on the assumption that the equipment of the companies is used, an average verification fee of £3 per wagon would enable the local authorities in whose areas the companies' calibration depots are situated to cover almost entirely the cost of the service.

169. Bulk sales of fuel oils are not the only sales which are effected by this form of measurement. Rail and sometimes road deliveries of milk take place in tank wagons in which the quantities are assessed by means of a calibrated dipstick; and the Milk Marketing Board has said that it would be desirable for these to be verified and stamped by inspectors. Here again, these instruments are undoubtedly in use for trade; and we recommend that the appropriate steps be put in hand to bring them under official control. This type of measurement can also be used for deliveries of beer to the vaults of licensed premises, although we gather that at present sales from tank wagons are mainly based on the quantity recorded in calibrated receptacles in the vaults themselves; but wherever this form of bulk measuring instrument may come to be used in trade for determining the quantities of liquids delivered, we consider that it should be subject to full Weights and Measures control and that suitable regulations should be made by the Board of Trade.

Liquid Fuel and Oil

370. The requirements at present applicable to the sale of liquid fuel and lubricating oil are contained in the Measuring Instruments (Liquid Fuel and Lubricating Oil) Regulations, 1929, which govern the construction and accuracy of measuring instruments used for individual deliveries not exceeding 20 gallons; and, under certain supplementary provisions, penalties can be imposed for the fraudulent operation of these measuring instruments. The associations representing retail petrol dealers and petrol manufacturers have stated that no serious difficulties have arisen in the enforcement of these regulations.

It has, however, been suggested to us that manufacturers and wholesalers would benefit if measures in excess of 64 gallons, which is the largest lawful measure at present, could be legalized; and this has been provided for in our recommendations on lawful weights and measures.

371. Petrol is normally sold both retail and wholesale by measure, whilst lubricating oils are sold either by measure or by weight, with the exception of the small amounts delivered from the grease-gun type of instrument which are normally retailed on a price basis only. There is, however, no legal obligation upon traders to sell by volume or weight; and the giving of short weight or measure is not in itself an offence. As already mentioned, we have received from retailers strong expressions of dissatisfaction at the absence of any Weights and Measures control over the wholesale deliveries of petrol; and we consider that our previous recommendations on the verification of tank wagons would remove the main obstacle in the way of applying short weight and measure provisions to this trade. We therefore recommend that all sales of liquid fuel and lubricating oil should be by net weight or measure only, that the Board of Trade should, in consultation with the interested parties, prescribe a lower exemption limit so that the small quantities of lubricating oil not normally sold by weight or measure can be excluded from this requirement and that the giving of short weight or measure should be an offence. We further believe it to be essential for adequate enforcement that, in wholesale deliveries of liquid fuel, each consignment should be accompanied by a delivery note stating the amount to be delivered, on the lines of the weight ticket at present required in deliveries of coal.

★ ★ ★

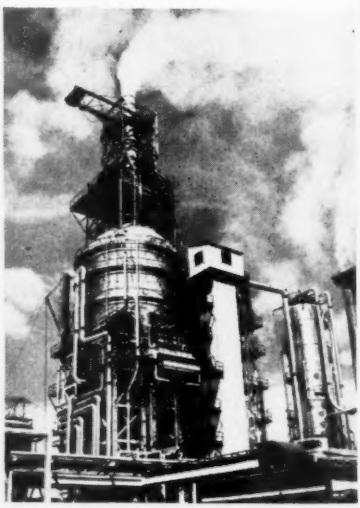
PERNIS CAT-CRACKER COMPLETED

The first modern-type catalytic cracking unit to be constructed on the European continent is now ready to come into full operation at Shell's Pernis refinery, near Rotterdam. Capacity of this new cat-cracker, which took nearly two years to build, will be about 1¼ million tons/year.

Since 1945 some £20 million has been spent on rehabilitation and new construction at the Pernis refinery, of which sum the new cat-cracker has absorbed a large part. Total capacity of the refinery has now been raised from the 1946 level of 1 million tons/year to 5 million tons/year.

Pernis has 19 jetties for tankers, a total charging capacity of 6000 cubic metres/hr, and a storage capacity of 1,200,000 tons.

The refinery employs 2700 persons.



Part of the cat cracker. The white rectangular structure in the front is the regenerator with the lift. The fractionating column can be seen to the right.

MODERN TECHNIQUES IN PETROLEUM TESTING

III. GAS ANALYSIS—OTHER THAN BY LIGHT ABSORPTION METHODS

By K. R. GARRETT*

In the last ten years, more attention has been focused on the utilization of petroleum derived gases than ever before. In the wake of this interest, methods of analysis applicable to gases have been developed to a very high degree.

This article aims at describing some of the more recent developments in the separation and estimation (other than by light absorption methods) of hydrocarbons having from one to six carbon atoms in the molecule, together with the estimation of some of the more commonly occurring impurities.

Much effort has been directed towards the production of automatic instruments for gas analyses. This has been particularly the case in the U.S.A., where instruments are now available for the estimation of small amounts of hydrocarbon gases in air, for the separation and analysis of gaseous mixtures, and for the estimation of small amounts of moisture, oxygen, hydrogen sulphide, etc., as impurities. Such equipment tends to become rather complicated and often requires to be serviced by a skilled mechanic.

Inflammable Gas Detectors

As a protection against explosion or fire, it is often necessary to analyse atmospheres for the presence of light hydrocarbon vapours, e.g., in buildings or ships where these inflammable vapours may accumulate. For this purpose, a number of combustible-gas detectors have been developed,[1] both as hand instruments and as continuous recorders. These function, in the main, by burning the hydrocarbons on the surface of a helical platinum filament. The change in resistance of the filament, brought about by its increase in temperature following the combustion, is a function of the concentration of the inflammable gas.

To correct for voltage variations and changes in atmospheric conditions, the Poole Explosimeter[2] employs two filaments mounted in a single cell. One acts as a catalyst and the other as a compensator. It is claimed that the instrument will detect 6 parts of propane or 4 parts of butane in 10,000 of air.

Low Temperature Distillation

The most widely used method for separating mixtures of petroleum gases is that of low temperature distillation. By this means the paraffins, methane to hexane, and paraffin-olefin mixtures up to C_3 hydrocarbons, may be separated into their individual components. Of the latter, those having 4, 5, or 6 carbon atoms in the molecule are usually separated into the three groups which may be further analysed by combustion and spectroscopic methods.

The apparatus used consists essentially of a distilling bulb provided with a heater, connected to a vacuum jacketed fractionating column containing a highly efficient packing, which is in turn connected to a receiver capable of working under reduced pressure. The top of the column is provided with a reflux cooling unit operated with liquid nitrogen (boiling point,—195.8°C) as coolant. The column head temperature is recorded by means of a single thermocouple and a suitable form of potentiometer. One of the most recent developments is an electronic type continuous balance recording potentiometer. A typical assembly is shown in Fig. 1.

*Calor Gas (Distributing) Company.

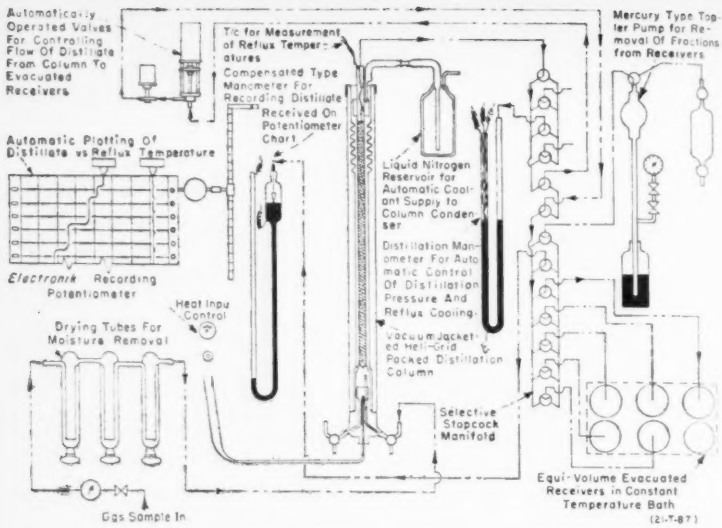

Automatically Operated Valves For Controlling Flow Of Distillate From Column To Evacuated Receivers
T/c for Measurement of Reflux Temperatures
Compensated Type Manometer For Recording Distillate Received On Potentiometer Chart
Mercury Type Toepler Pump for Removal Of Fractions from Receivers
Automatic Plotting Of Distillate vs Reflux Temperature
Liquid Nitrogen Reservoir for Automatic Coolant Supply to Column Condenser
Distillation Manometer For Automatic Control Of Distillation Pressure And Reflux Cooling
Electronik Recording Potentiometer
Heat Input Control
Drying Tubes For Moisture Removal
Vacuum Jacketed Helix Packed Distillation Column
Selective Stopcock Manifold
Equi-Volume Evacuated Receivers in Constant Temperature Bath
Gas Sample In

Fig. 1. Flow diagram for Podbielniak low-temperature distillation apparatus.

As carbon dioxide, hydrogen sulphide and water vapour might freeze out and block the column, they are first removed from the sample by passing it through a soda-lime scrubber followed by a tower packed with a suitable drying agent, e.g., barium oxide, anhydrous calcium sulphate, etc. The gases are then charged into the distillation bulb where they are cooled by means of liquid nitrogen. Providing this procedure is carried out at a suitable rate, uncondensable gases such as hydrogen, nitrogen, oxygen, carbon monoxide, and part of the methane, may be taken overhead from the distillation column during charging.[3] This fraction may be analysed by absorption and combustion methods. Thomas *et al*[4] have described a method for the removal of the uncondensed gases by means of a Toepler pump. After the uncondensed gases have been taken off, the heating in the distilling bulb and the cooling at the top of the column are adjusted so that the column is maintained under total reflux for a definite period.

When equilibrium conditions have been reached, the gaseous fractions are taken off. Throughout the distillation the column is maintained at a constant pressure, usually 300 mm for hydrocarbons up to the butanes, 100 mm for pentanes, and 50 mm for hexanes. This is effected by balancing the heat input to the distilling bulb against cooling at the top and against the rate of withdrawal of distillate.

With certain types of apparatus it has been found that distillation rates as high as 100 c.c. per minute may be allowed with negligible contamination by lower boiling fractions.[5] The boiling point of each fraction is plotted against the volume of distillate collected. The latter may be measured by salt water displacement or, more usually, by the increase in pressure of constant volume evacuated receivers situated in a thermostatically controlled bath.

Often a high boiling hydrocarbon called a 'chaser' is added to the liquefied gas in the distilling bulb. During the distillation this transfers heat from the distilling bulb to the sample with consequent improvement in separation, reduction in the time of distillation, reduction in the amount of sample required and allows complete removal of the C_4 fraction from the dead space in cases where the C_5 fraction is very low. It is important to ensure, however, that the chaser will not form an azeotropic mixture with any of the constituents of the gas.

W. G. Podbielniak[6] has been one of the pioneers in the development of laboratory distillation apparatus. The latest design of Podbielniak Inc. includes a column which allows part of the liquid nitrogen vapours to pass down part of the annular space between the column and the jacket. This arrangement compensates for heat leakage through the vacuum jacket and for the heat capacity of the packing and inner parts of the glass column. With the Buta-Heli-Grid wire coil packing *trans*-butene-2 (B.P. 0.9°C) has been satisfactorily separated from the *cis* isomer (B.P. 3.7°C).

It is also possible to pass a stream of dry air up the annular space. This useful feature reduces markedly delays that would otherwise occur after each break due to the heat capacity of the column and also is of assistance in warming up the column towards the end of a distillation, when no liquid is left in the distilling bulb.

The latest model foresees when a change in distillation composition is about to occur; it automatically sets the distillation rate to that required for optimum performance; it produces a record of the distillation analyses; it is self-checking and even signals for aid when necessary, e.g., when the liquid nitrogen requires replenishing or when the limiting pressure in the receiver has been reached.

A simplified form of apparatus employs a silvered vacuum jacketed column packed with small open-ended cylinders of stainless steel gauze.[7] A semi-automatic control of distillation rates may be effected by using a manometer in the column system to control the flow of nitrogen to the cooler. Douslin and Walls[8] have described a more complicated method of controlling the flow of cooling medium by means of a throttling valve operated by the manometer.

Information on a proposed method of analysis of liquefiable hydrocarbons by distillation was published by the IP Panel in 1945.

Mass Spectrometer

The mass spectrometer,[9,10] developed during the late war for qualitative and quantitative gas analyses, has been described in a previous paper.

Gas mixtures containing up to nine components[11] may be analysed with greater speed than by low temperature distillation and chemical absorption tests. It is of particular value in control testing laboratories where a large number of samples are required to be analysed in the shortest possible time. It has been used successfully for detecting low concentrations of impurities in relatively pure gas streams. Since the pressure maintained inside the instrument is very low, only very small samples of around 0.2 to 0.4 ml (measured at atmospheric pressure) are required.

According to C. E. Starr and T. Lane,[12] reporting on the work of 70 laboratories, its accuracy is about the same as that of low temperature distillation for total C_3, total C_4, and total C_5 hydrocarbons. The probable error was 0.2 to 0.5 mole per cent for C_4 components, except for *iso*butane and total *n*-butenes, when it was nearer 1.0 per cent. To overcome the lack of sensitivity in estimating isomers, it has been successfully used in conjunction with the infra red spectrometer.[13]

Adsorption Fractionator

An adsortpion fractionator, employing a column of activated charcoal,[14] has been used to a limited extent in the U.S.A., for the separation of simple gaseous mixtures. As the gases are introduced in the base of the column, the lighter ones, having less relative adsorption, pass farther up the column than the heavier ones which tend to concentrate at the bottom.

A feature of this apparatus is that the heater is slowly raised up the column as fractionation proceeds. Although no more rapid in use than a Podbielniak apparatus, the adsorption fractionator does not require liquid nitrogen for its operation, a factor of some advantage in isolated locations.

Mercury, vaporized by means of a heater, plays the role of stripping agent. Gas in the heater is desorbed and driven upwards. The heavier gases increase in concentration in the zone just ahead of the mercury vapour, whilst the lighter gases gain in concentration in the zone above until a separation is obtained. The column ends in a section of small diameter to increase the sharpness of separation.

It is claimed that the apparatus will separate hydrogen, carbon dioxide and hydrocarbons into groups up to C_6 but the time taken is around 7 to 9 hours. The fractions are identified by means of thermal conductivity measurements.

Absorption Methods

A novel method of gas absorption analysis has been described by Gooderham.[15,16] Gas, free from dust and fog, is passed into an apparatus consisting of soap bubble meters and reagent vessels connected in series. Each vessel contains a reagent to remove one constituent so that, when the system is in equilibrium, the difference between the readings of two consecutive meters indicates the amount present in the gas of the constituent removed by the intervening reagent vessel.

The whole apparatus is specifically designed to give no appreciable back pressure and, where possible, solid absorbents are used. The number of reagent vessels is decided by the number of gaseous components it is required to remove. In general, it is necessary to pass one litre of sample for equilibrium to be obtained. Accuracy claimed is 0.2 per cent on each component.

Two recent improvements in the fabrication of general apparatus for absorption analysis are worthy of mention. The first is the use of ball and socket joints which are now widely used in the U.S.A. These are more flexible than taper joints and consequently reduce the risk of breakage. Rubber tubing connexions on Orsat apparatus, etc., may be completely eliminated by the use of these joints. Their manufacture in the U.K. is just beginning.

The second is the use of strip metal pieces which facilitate rapid assembly of all types of framework for holding apparatus, Such supports may be dismantled when not required and the pieces used afresh.

Determination of Unsaturated Hydrocarbons

For the determination of unsaturated hydrocarbons by absorption it is necessary to choose the reagent which will give the optimum performance. This will depend on the type of gas to be analysed, the apparatus to be used and the accuracy required.

To reduce errors such as those due to reaction of the absorbents with paraffins, the solubility of the paraffins in the reagents and the alkylation of olefins with paraffins, many methods have been modified in recent years.

For the determination of total olefins one reagent is a 22 per cent solution of mercuric sulphate in 22 per cent sulphuric acid, described by Francis and Lukasiewicz.[17,18] Carbon dioxide is slowly absorbed by this reagent and should therefore be removed by first

passing the gas into caustic potash solution.

A 3.5 per cent solution of silver sulphate in 72 per cent sulphuric acid[19] gives good results for certain mixtures of gases but is not very satisfactory where there is a high ethylene content, as the absorption of ethylene is reversible. If the solution has been in use for some time, it is possible for the ethylene to be desorbed into the sample when a gas low in ethylene is being examined.

Various concentrations of bromine solutions have been used for many years but many of them react with the paraffins, particularly with the butanes. Savelli *et al*[20] claim that the use of one-fourth saturated bromine solution (containing 50 g per litre of potassium bromide) produces results comparable with other methods. Unlike the more concentrated solutions, this strength of bromine does not appreciably attack the butanes.

A simple method for laboratory routine work[21] involves dissolving the gaseous hydrocarbons in cold chloroform and titrating the C_3, C_4, and C_5 olefins with a solution of bromine in glacial acetic acid. Ethylene does not react if present in concentrations less than 10 per cent. 1.3-Butadiene is so slowly brominated that erratic results are obtained. It should, therefore, be removed by maleic anhydride prior to the analysis. Hydrogen sulphide and mercaptans interfere and should also be removed before analysis by scrubbing with doctor solution.

A satisfactory separation of *iso*butene and butene-1 is practically impossible by low temperature distillation because their boiling points differ by only 0.6°C. It is therefore usual to estimate *iso*butene in C_4 fractions by one of three chemical methods.

The first employs 60-70 per cent sulphuric acid as the absorbing reagent. This absorbs some of the normal butenes and butadiene so that it is necessary to apply correction factors for this absorption.

The second depends on the reaction between hydrogen chloride and *iso*butene with the formation of tertiary butyl chloride. Other C_4 and C_3 hydrocarbons do not react but several of them are soluble in the aqueous reagent. Houtman[22] has investigated some of the corrections to be applied. He reports that the solubility of butadiene and butene is as high as 19.0 ± 0.8 ml and 8.5 ± 0.4 ml respectively, in 100 ml of the reagent (1/1 HCl saturated with NaCl) at 20°C and atmospheric pressure.

The third using Denigès reagent (mercuric oxide in nitric acid) is probably the best of the three. When *iso*butene is passed into this reagent it is absorbed. On boiling the solution, a yellow precipitate, of somewhat doubtful composition, is formed. Newton and Buckler[23] found that this is slightly soluble in the nitric acid solution and that its composition changes slightly when washed with water. They overcame these difficulties by neutralizing the reagent with caustic soda and determining the mercury content of the precipitate. Under these conditions the ratio of *iso*butene to mercury in the precipitate was found to be constant at one molecule of *iso*butene to seven atoms of mercury. The mercury may be estimated by dissolving the precipitate in hot 70 per cent nitric acid and titrating with standard potassium thiocyanate solution using ferric alum as indicator.

1.3-Butadiene and normal butane form a constant boiling mixture so that they cannot be separated by low temperature distillation. However, butadiene may be determined by absorption in molten maleic anhydride.[24] If large quantities of *iso*butene are present, poor results are obtained, possibly due to the polymerization of the *iso*butene in the molten absorbent. To reduce this effect, Elving and Stein[25] added 2 per cent of diamylamine to the maleic anhydride to act as an inhibitor and Roman[26] first

reduced the *iso*butene content of the gas to below 15 per cent by passing it through 50 per cent sulphuric acid.

Several hydrocarbons are soluble in maleic anhydride but the effect may be minimized by saturating it with the gas under test before the actual determination is carried out. This practice applies equally for all the reagents mentioned above.

Another method for reducing the effect of physically absorbed unreacted gases is to sweep them out with carbon dioxide. Any excess carbon dioxide may be removed by caustic soda before the residual gas is measured. This only applies when the reagent does not react with carbon dioxide, e.g., in the maleic anhydride mentioned above.

The subject of gas analysis by absorption has been discussed by the Liquefied Gases Panel of the IP.[40]

Oxygen

Oxygen may be satisfactorily determined in hydrocarbon gases by taking advantage of its paramagnetism. Nitric oxide, nitrogen peroxide, and chlorine dioxide are also paramagnetic but these gases are not likely to be present. A wire in a non-uniform magnetic field undergoes cooling when a paramagnetic gas surrounds it. The amount of cooling is a function of the concentration of the paramagnetic gas present. The Distillers Company and the Hays Corporation (U.S.A.) have recently developed improved models of oxygen analysers based on this principle.

Several chemical methods have been developed and several improved recently, particularly for the estimation of low concentrations of oxygen.

The method due to Shaw[27] gives good results for concentrations over 0.001 per cent. A known volume of gas is shaken with freshly precipitated ferrous hydroxide (oxygen free) in a special apparatus for approximately one hour, until absorption is complete. If unsaturated hydrocarbons are present in the gas, they should be removed at this stage by evacuation. The solution is then acidified and the ferric iron formed is determined colorimetrically by the thiocyanate method. Hydrogen sulphide and volatile bases which interfere may be removed by scrubbing with alkali and acid.

Winslow and Liebhafsky[28] found that the Winkler method could be used for oxygen concentrations of 0.001 to 0.1 per cent. The oxygen is absorbed by shaking the gas with manganous hydroxide. On acidifying the oxidized solution in the presence of potassium iodide, an equivalent amount of iodine is liberated. This may be compared with standard colour solutions or titrated with standard thiosulphate solution using starch as indicator.

The basis of another method, which is not affected by the presence of olefins and diolefins, depends on the reaction between copper and oxygen in ammoniacal solutions.[29,30] An apparatus, containing a reaction cell packed with copper ribbon, is purged with nitrogen to remove all traces of air. The copper ribbon is cleaned free from oxide by washing with several portions of ammonia—ammonium chloride solution. The cell is partly filled with this reagent and a known volume of the sample is passed in. All the ammoniacal solution is run out, collected, and the majority of the ammonia is boiled off. After cooling, the solution is acidified with acetic acid. The copper content, equivalent to the oxygen absorbed, is estimated by the addition of potassium iodide and by titrating the liberated iodine formed with thiosulphate in the usual manner. Although the method is suitable for concentrations from 0.001 to 5 per cent, higher concentrations may be estimated by suitably diluting the sample with nitrogen. Hydrogen sulphide, mercaptans, acetylene, and sulphur dioxide interfere and should be removed by successive scrubbing with aqueous silver nitrate and caustic potash pellets.

Powell and Joy[31] have designed a simplified portable form of the apparatus which is more suitable for plant work. The intensity of blue colour, due to the solution of copper, is determined optically.

MOISTURE

Physical Methods.—The estimation of water in gases by means of the dew-point method is limited to non-corrosive gas mixtures having a hydrocarbon dew-point at a lower temperature than the water dew-point.[32] If the higher hydrocarbons condense on the mirror surface first, it is impossible to detect the formation of the water deposit with any degree of accuracy.

A continuous dew-point recorder has been developed by the General Electric Co. (U.S.A.). The mirror is cooled by a refrigerant in the usual manner but the light reflected from the mirror falls on a photoelectric cell which controls the output of a small heater near the mirror. In this way a steady cycle between dew and no dew can be obtained, the maximum amplitude of which is claimed to be 2°F.

Weaver and Riley[33] have developed a method employing a thin film of hygroscopic material, e.g., phosphoric acid with gelatine as a binder, spread over the surface of a solid insulator situated between metal electrodes. When surrounded by the gas under test, the water vapour of the film changes until it is in equilibrium with the partial pressure of the water vapour in the gas. It was found that the change in electrical conductivity of the film was proportional, over a wide range, to the moisture content in the gas. The moisture content of a sample is obtained by comparison with a gas containing a known amount of water. The pressures of the sample and standard are adjusted until they both have the same water vapour content, as shown by the production of an equal resistance of the detecting film. Moisture standards are obtained by saturating air, oxygen or nitrogen at about 35 atmospheres and allowing the gas to expand to atmospheric pressure.

In a similar manner, the recording electrical hygrometer of the American Instrument Co., employs a coil of wire coated with a film of partially hydrolysed polyvinyl acetate, containing a small proportion of lithium chloride. The change in resistance of the coil is used as a measure of the amount of water in the film and hence the humidity of the surrounding gas.

Absorption and Chemical Methods.—Absorption methods have not yet been developed to a completely satisfactory stage for accurate determinations. All the solid absorbents tried to date, take up light hydrocarbons as well as moisture. Cappell *et al*[34] have found that unsaturated compounds such as ethylene and propylene are polymerized when in contact with certain solid absorbents and that these polymerized products remain behind in the drying agent. However, many workers have attempted to reduce errors by first saturating the absorbent, e.g., anhydrous magnesium perchlorate,[35,36] with the gas under test.

Levin *et al*[37] claim that as little as 0.001 per cent of moisture may be determined by an acetyl chloride hydrolysis method. The water is removed from the sample by contact with dry acetone and pyridine, maintained at about −60°C. The acetone-water mixture is taken off, reacted with acetyl chloride and the liberated acid is titrated with standard alkali. Many workers have tried this method but some have found difficulty in obtaining good results.

The use of Fischer reagent (a solution of iodine, sulphur dioxide and pyridine in methanol) has shown great promise. Instead of reacting the acetone-water solution with acetyl chloride as above, Mitchell and Smith[38] titrated it with Fischer solution to the visual end-point. Good results were obtained in the

writer's laboratory by passing the whole of a sample of gas through excess Fischer reagent contained in a titration cell. The excess reagent was back titrated with standard methanol-water solution. The end-point may be determined colorimetrically as a change from dark red to yellow or electrometrically. The gas passed may be obtained by weighing the sample vessel before and after the test or by means of a gas meter placed after the cell. If a wet meter be used, a moisture trap should be inserted between the cell and the meter. If mercaptans are present in the sample and no hydrogen sulphide is present, they should be determined separately, say by the silver nitrate method,[39] and the necessary correction applied.

For the determination of moisture in liquefied hydrocarbons, it is preferable to take a special sample and pass the whole of it through the reagent because the vapour phase contains a higher proportion of water than the liquid phase.

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★ ★ ★

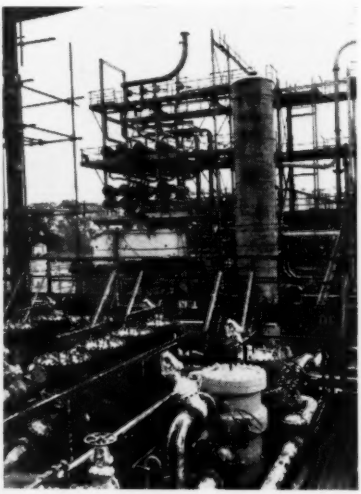
Synthetic Detergents. A new disk for the determination of synthetic detergents is described in a leaflet issued by The Tintometer Ltd., Salisbury, titled "The estimation of anionic synthetic detergents in sewage using the Lovibond comparator."

"THE DIESEL STORY"

This new Shell film is designed as an introduction to the study of the modern diesel engine and therefore the subject is treated in a manner suitable for a wide variety of audiences. The history and development of the internal combustion engine is traced from the first "Otto" gas engine of 1877, the Ackroyd Stuart "hot bulb" engine of 1890, the first experimental "diesel" built at Augsberg in 1893, to the first reliable diesel in 1897. By 1900 the diesel engine was in regular production and in 1912 the first ocean-going diesel ship—the *Selandia* of 7400 tons—was launched.

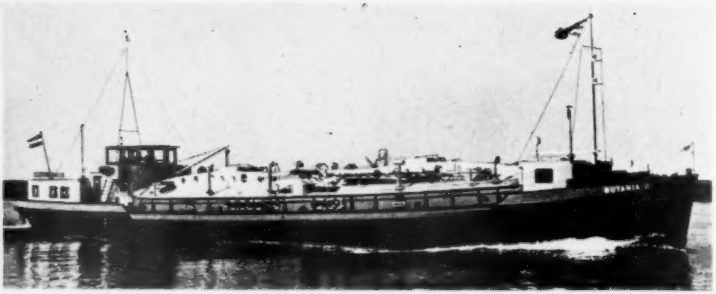
Since then developments have been great, and to-day the diesel is in large scale production throughout the world in all types and sizes.

The film is available in 16 and 35 mm sizes and runs for 18 minutes.



This close up taken at Anglo-Iranian's new Kent oil refinery, Isle of Grain, shows the heat exchanger equipment on the distillation unit with valves for adjusting oil flow.

An X-ray machine is being used to examine a locally welded seam on a fractionating column at the Kent oil refinery now under construction. This machine will detect flaws in plating up to several inches thick.



Butania II is one of the first two lighters in Europe to be specially designed for the bulk carriage of liquid petroleum gas. These two vessels, each with a capacity of 125 metric tons, ply between Pernis refinery in Rotterdam and Ghent and Brussels, where there are large demands for L.P.G. for domestic and industrial purposes. The lighters are owned and operated by N.V. Internationale Riviertankscheepvaart Mij, a Shell associate.

"OIL FOR THE 20th CENTURY"

Originally designed by Anglo-Iranian to celebrate the 50th anniversary of the 1901 D'Arcy agreement, this fine documentary film covers within the running time of half-an-hour the exciting story of the search for oil in Persia.

Produced by the Pathé Documentary Unit, the film is a kind of scrapbook, for it contains sequences from old news reels and much footage shot by Anglo-Iranian cameramen during the past 30 years in Persia, as well as new material. All this is smoothly welded together, and the film is greatly assisted by the excellence of its running commentary. There are two narrators, the one giving the general descriptions having a standard "B.B.C." voice, while a persuasive Scots accent tells, in the first person, the story of field operations.

But attractive as are the shots of the up-to-date installations of Abadan, and of the facilities and amenities for staff, there is peculiar fascination in the sequences taken from old films. For example, there are early shots of pipe laying, where the operators appear to work their primitive apparatus in a series of jerks, and loaded donkeys seem to trot uphill at a great pace; of London in the first years of the 20th century, when "horsepower" meant the power of a horse; a glimpse of the young Churchill; and an amusing sequence of the old Abadan refinery fire brigade, reminiscent of an early Chaplin comedy.

"Oil for the 20th Century" has had a West End presentation in London, and is being shown throughout the provinces in A.B.C. cinemas.

★ ★ ★

LONDON BRANCH DANCE

Following their highly successful function last year, the London Branch have decided to organize another buffet dance at Manson House on Friday November 16, 1951. Eric Lambert's orchestra will again provide the music.

Dancing is from 8 p.m. until midnight, and tickets are 10/- each. Members of the Institute will be very welcome, and tickets can be obtained from the General Secretary at Manson House or from London Branch Committee members.

There will be a running buffet, which is included in the price of the ticket, and a licensed bar.

Evening dress will be worn. Early application for tickets is advised, as accommodation is limited.

FORTHCOMING MEETINGS

THE INSTITUTE

Detergents from Petroleum. S. F. Birch, Ph.D., (*Anglo-Iranian Oil Co.*). At 26 Portland Place, London, W.1, 5.30 p.m. (*tea 5 p.m.*), November 14.

Methods for Expressing the Viscosity-temperature Relationships of Lubricating Oils. F. T. Blott, B.Sc., ("*Shell*" *Refining & Marketing Co.*) and C. G. Verver, B.Sc., (*Koninklijke/Shell Laboratorium, Amsterdam*). At 26 Portland Place, London, W.1, 5.30 p.m. (*tea 5 p.m.*), November 29.

Pre-flame Reactions in a Diesel Engine. Prof. F. H. Garner, O.B.E., Ph.D., Prof. F. Morton, Ph.D., and W. D. Reid (*University of Birmingham*) and W. E. Malpas (*Trinidad Leaseholds Ltd.*). At 26 Portland Place, London, W.1, 5.30 p.m. (*tea 5 p.m.*), December 12.

FAWLEY BRANCH

Concepts of Industrial Training. N. F. Myers. At Esso Recreation Club, Holbury, 8 p.m., October 17.

Search for Oil in England. Film introduced by R. K. Dickie. At Esso Recreation Club, Holbury, 8 p.m., November 14.

LONDON BRANCH

Aircraft Gas Turbine Fuels. J. G. Dawson. At 26 Portland Place, London, W.1, 6 p.m. (tea 5.30 p.m.), October 24

People and Oil. A. C. Palmer. At 26 Portland Place, London, W.1, 6 p.m. (tea 5.30 p.m.), November 20.

NORTHERN BRANCH

The Law and Petroleum. V. Biske, LL.M., B.Sc. At Engineers' Club, Albert Square, Manchester. 6.30 p.m., October 16.

The Petroleum Chemical Industry of N.W. England. Dr H. K. Whalley, B.A. At Engineers' Club, Albert Square, Manchester. 6.30 p.m., November 20.

SCOTTISH BRANCH

Construction of a Modern Oil Refinery. W. Robb and W. M. Greenhorn. Edinburgh, November 1.

STANLOW BRANCH

World Petroleum Development. C. A. P. Southwell, M.C., B.Sc. At Grosvenor Hotel, Chester. 7.30 p.m., October 17.

Industrial Applications of Spectroscopy. J. G. Reynolds. At Grosvenor Hotel, Chester. 7.30 p.m., November 21.

OTHER SOCIETIES

Reactions of Hydrocarbon Radicals. Prof. C. E. H. Bawn, Ph.D. The Chemical Society and the Werner Society, at Chemistry Department, Trinity College, Dublin, 8 p.m., November 9.

Discussion on the Application of Statistical Methods to Chemistry. Royal Institute of Chemistry (London & S.E. Counties Section). At Waldorf Hotel, London, W.C.2., 4 p.m., October 25.

Separation of Gases by Low Temperature Methods. J. B. Gardner, B.Sc., Ph.D. Royal Institute of Chemistry. (London & S.E. Counties Section). At Acton Technical College, London, W.3. 7.30 p.m., November 29.

Some Fuel and Power Projects. H. Roxbee Cox, Ph.D. Institution of Mechanical Engineers. At Storey's Gate, London, S.W.1., 5.30 p.m., November 16.

Melchett Lecture. Prof. F. H. Garner, O.B.E. The Institute of Fuel. At The Institution of Mechanical Engineers, London, 5.30 p.m., (*tea* 5 *p.m.*), October 16.

Fuel Oil in Industry. T. O. Ambrose, The Institute of Fuel (Midland Students' Section). At The University, Edmund St., Birmingham. 7.30 p.m., November 12.

Roads of the Past—their Makers, Users, Traffic and Mode of Construction. Prof. R. J. Forbes. Society of Chemical Industry (Road and Building Materials Group). At Institution of Structural Engineers, Upper Belgrave Street, London, S.W.1., 6 p.m, November 15.

PETROLEUM INDUSTRY EXHIBITION

The response to the Exhibition of the Petroleum Industry recently shown in London, Glasgow, Leeds and Cambridge has led to its being invited to three other University towns.

Therefore, it will be on view at:

Leicester (Museum and Art Gallery, October 8-27).

Sheffield (Firth Hall of the University, November 8-17).

Edinburgh (Royal Scottish Museum, December 17-January 27).

At Sheffield it will form the background to a series of lectures on the petroleum industry,

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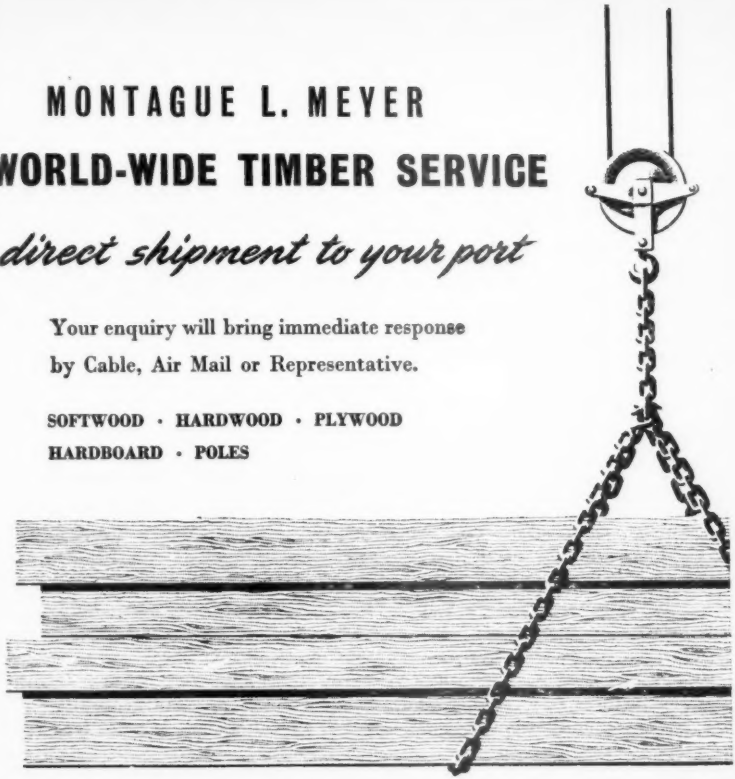

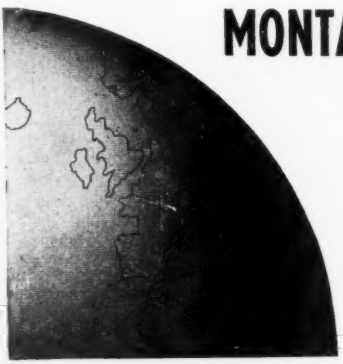

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Refinery Manager—Minimum age 40. Chemical or Mechanical Engineer preferred. Candidate must have had approximately 20 years' experience in Refinery operations, including administrative functions. Past record must indicate capacity to hold important position with major Oil Company, ability to handle people and to co-ordinate the work of a large supporting staff. The successful applicant will be responsible to the Director of Manufacturing for the overall performance of the Refinery.

Assistant Refinery Manager—Will act as deputy to Refinery Manager and must possess similar qualifications and have had similar, if slightly less, experience. Minimum age 35. The successful applicant will be chosen for his potential to progress in the Company as well as for his capacity for this position.

Operating Superintendent—Will be responsible to the Assistant Refinery Manager for the Operating Division. Preferably a Chemical Engineer. 15 years' practical experience in Refinery operations essential. Aged between 35 and 50.

Chief Engineer—Will be responsible for the mechanical engineering phase of the operating Refinery. Candidates must possess an Engineering degree. Minimum age 35. An essential requirement is a background of 10 to 15 years' practical experience directly or indirectly associated with Refinery engineering. Particular emphasis is laid on ability to work in a staff capacity with other people.

Utilities Department Manager—Candidates must possess a Mechanical or Electrical Engineering degree or equivalent. Minimum age 35. A minimum of 7 years' experience in power plant (400 p.s.i. or higher) operation considered essential. Will be responsible to the Operating Superintendent for the operation of the power plant and the distribution of all utilities at the Refinery. Ability to handle people considered to be an important factor.

Corrosion or Inspection Engineer—Engineering degree or equivalent. Minimum age 35. At least 5 years' previous experience of boiler or pressure vessel inspection, preferably in a Refinery. Will be responsible for inspection of all Refinery equipment, interpretation of results, recommendations of material specifications. Ability to work with others considered essential.

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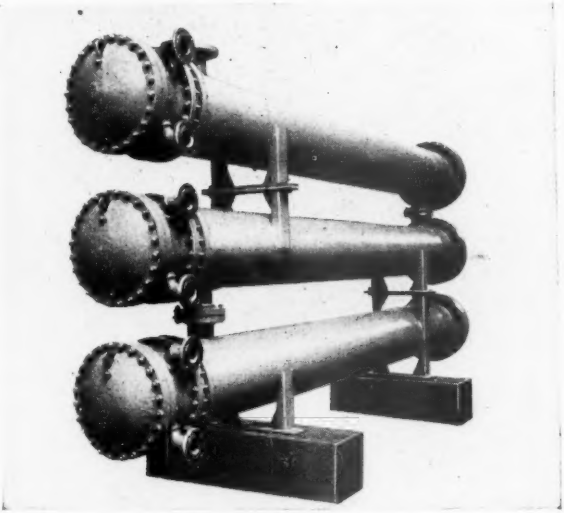

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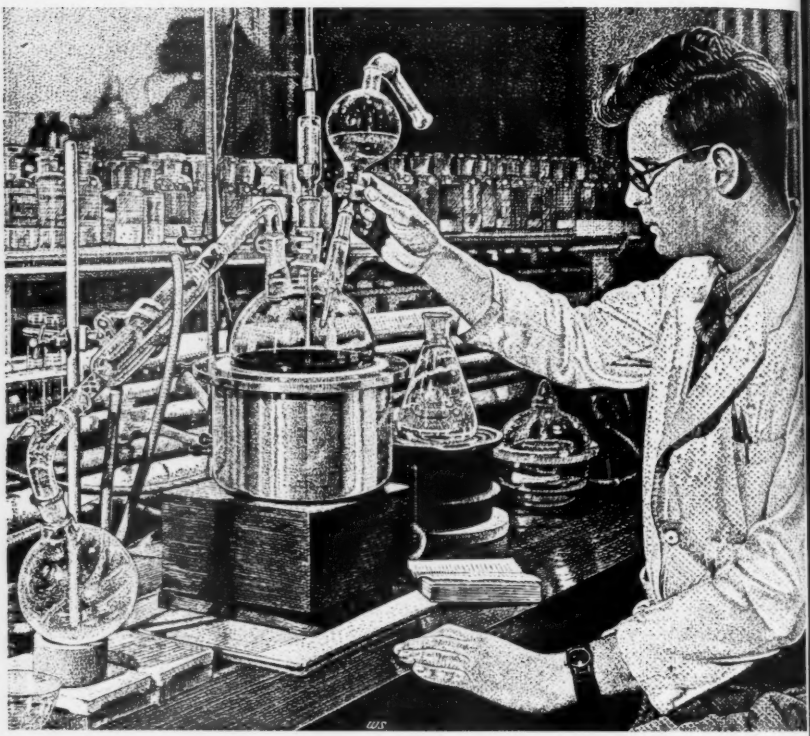

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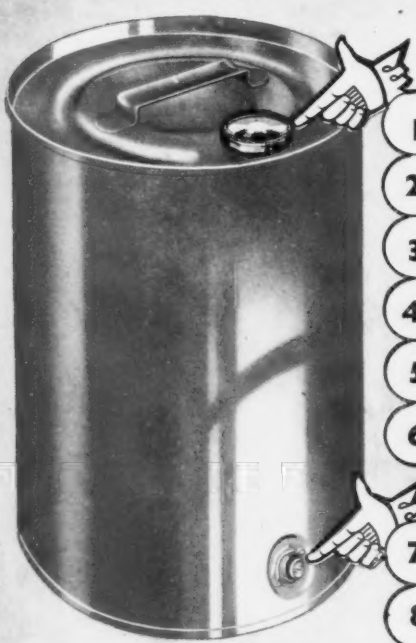

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